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Thermal Stability of Six Candidate Propellants for an Air-Launched Tactical Missile

by
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FEBRUARY 1982

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FOREWORD

This report describes calculations using data from tests of six candidate propellants to determine their thermal stability under selected hazardous environments. The studies were performed during the time period November 1980 through March 1981.

The effort reported herein was performed by the Naval Weapons Center (NWC) and supported by the Naval Air Systems Command under AirTask A03W3300/008B/1F31300000. This report was reviewed for technical accuracy by Ronald F. Vetter.

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(U) The thermal stability patterns of six solid propellants of various types were determined by means of differential thermal analysis, thermogravimetric analysis, and derivative thermogravimetry. Differential scanning calorimeter data were also obtained. These data were then used to calculate the time to cook-off of the propellants. Examples show the necessity of considering self-heating effects when the heating times are long (i.e., slow cook-off rather than fast cook-off). The methods and techniques described can be used to predict cook-off time and temperature for other propellants.

CONTENTS

INTRODUCTION

Methods and techniques are needed for predicting the temperature and time to cook-off of solid propellants in an air-launched tactical missile, taking into account the self-heating events that can be generated within these propellants. The topic of propellant ignition within an air-launched tactical missile under selected hazardous environments has previously been addressed by Hercules Incorporated.[1] The thermal hazards considered by Hercules were such as might occur in a flight deck environment and included exposure to the impinging exhaust of a jet engine or of a huffer/jet engine starter. In addition, the effects of a fuel fire on the deck were considered. The purpose of the study reported herein was to determine and compare the thermal stability of six propellants, using several experimental techniques to define their autoignition temperatures, and then to calculate the self-heating events for these propellants. Finally, the data obtained were used in conjunction with the Hercules data of footnote 1 to evaluate the risk from the selected hazards. The methods and techniques developed can be used with any propellant to predict temperature and time to cook-off.

TEST APPROACH

PROPELLANTS

The six propellants studied were selected to cover a wide range of predicted autoignition temperatures. They also represent Class 1.1, marginal, and Class 1.3, hazard categories. Basically, they were of three different types:

1. Minimum smoke propellant (Class 1.1) containing a nitrate ester. Generally, a nitrate ester starts to thermally decompose sooner than ingredients contained in the other two propellant types; hence, these propellants are the least stable of those considered. The two chosen of this type were GBP-1 and GCV.

[1] Chemical Propulsion Information Agency. "Thermal Hazards of Turbine Exhaust Impinging on a Solid Rocket Motor," by R. B. Lening and R. L. Peterson, Hercules Incorporated. Presented at 1980 JANNAF Propulsion Systems Hazards Specialist Session, 13 March 1980. Laurel, Md., CPIA, May 1980, pp. 61-71. (CPIA Publication 319, document UNCLASSIFIED.)

2. Propellant containing both AP and HMX.* Such propellants are generally less thermally stable than propellants containing only one of these ingredients. The two propellants of this type were QBC (reduced smoke) and UTP-15908A (baseline).

3. A basic (Class 1.3) AP-rubber propellant, with and without aluminum. Of the three propellant types, these are the most thermally stable. The two propellants of this type were KAA-114 (baseline) and SAO-109 (reduced smoke).

Small samples of the selected propellants were supplied in the proper form for use in the test equipment. Detailed data on these propellants are given in Table 1.

EXPERIMENTAL METHODS

Laboratory tests were conducted on small samples of the selected propellants to determine their thermal patterns. A Mettler Instrument Company Thermoanalyzer-2 was used to obtain two simultaneous measurements: weight change and energy generation/absorption. Differential thermal analysis (DTA), thermogravimetric analysis (TGA), and derivative thermogravimetry (DTG) traces are produced by the Thermoanalyzer-2. The use and application of this technique were described in a previously published report.[2]

In another technique, a Perkin-Elmer Model 1B differential scanning calorimeter (DSC) was used to obtain thermal scan rate data. Details of this technique are also described in the footnote 2 report.

In addition to the laboratory tests performed by NWC, some DSC data on the propellant samples were supplied by Hercules Incorporated. The data, which were obtained on an apparatus similar to that used by NWC, are included in this report for comparison.

TABLE 1. Type and Composition of Candidate Propellants.

Type	Designation	Composition	Solids loading, wt.%	Plasticizer	
				Type	Wt.%
Minimum smoke	GBP-1	XLDB–HMX	59	NG	29
	GCV	XLDB–RDX	...	NG	...
Baseline	KAA-114	CTPB–AP–Al	87	DOA	3
	UTP-15908A	HTPB–HMX–AP–Al	90	...	...
Reduced smoke	QBC	HTPB–HMX–AP–Al	90	IDP	2
	SAO-109	HTPB–AP	87	IDP	3

* A Nomenclature is given at the end of this paper.

[2] Naval Weapons Center. *NWC Standard Methods for Determining Thermal Properties of Propellants and Explosives*, by Jack M. Pakulak, Jr. and Carl M. Anderson. China Lake, Calif., NWC, March 1980; 44 pp. (NWC TP 6118, publication UNCLASSIFIED.)

TEST RESULTS

In the discussions which follow, the temperature data appear to be inconsistent because the actual DTA, TGA, and DTG data are in degrees Celsius; the DSC data obtained at NWC are in Kelvins; and the data from Hercules are in degrees Fahrenheit, Celsius, and Kelvin. All calculations have been made in Kelvins and, when appropriate, converted to be consistent with the original data.* The data from any source are usually in their original units; a few data points have been converted to the predominant units (e.g., DTA data in Table 2).

The DTA, TGA, and DTG thermal patterns on the six propellants were determined at heating rates of 3 and 10°C/min. These thermal patterns are shown in Figures 1 through 13.

The thermal patterns for GBP-1 are shown in Figures 1 and 2. The onset temperature to the first weight loss reaction was 102°C; onset to the DTA exothermic action was 156°C, and the DTG peak was 180°C. The weight loss for the first exothermic peak was about 30% of the original weight of the sample.

The thermal patterns for GCV are shown in Figures 3 and 4. This propellant had an onset to the weight loss reaction of about 110°C and a DTA exothermic onset temperature at about 165°C. The temperature for the first exothermic peak (DTA and DTG) was 193°C. The second DTA and DTG peaks were at 224°C. The weight loss reaction for the first exothermic peak was not completely separate from the second peak, but it did account for about one-third of the sample weight. (These data were taken from Figure 3. The thermal data at the higher heating rate in Figure 4 does not distinguish two exothermic peaks.)

Thermal patterns for the QBC propellant are shown in Figures 5 and 6. The temperature for the initial or onset weight loss was 170°C. A small weight loss and DTG peak occurred at 195°C. A small exothermic peak also occurred at 202°C. For this decomposition area, the sample lost about 6% of its original weight. The main exothermic peak occurred at 245°C at a heating rate of 3°C/min.

The thermal patterns for the UTP-15908A propellant are shown in Figures 7 and 8, and a thermal pattern for an older sample of UTP-15908 propellant, made approximately 9 years ago, is shown in Figure 9. The onset temperature for the weight loss reaction was about 145°C for the UTP-15908A and about 160°C for the older UTP-15908 propellant. The onset temperature for the exothermic reaction was about

* If the reader requires temperature conversions, the following information is presented:

To convert from	To	Action
°C	K	°C + 273.15
°F	°C	(°F − 32)/1.8
°F	K	(°F + 459.67)/1.8

200°C with a peak temperature at 242°C, ru· at 3°C/min, and 250°C, run at 10°C/min, for the UTP-15908A.

The thermal patterns for KAA-114 are shown in Figures 10 and 11. The onset temperature to the weight loss reaction was 182°C, and the onset to the exothermic action was 210°C, with a peak temp erature at about 246°C.

The SAO-109 propellant (Figures 12 and 13) had an early onset to the weight loss reaction at 130°C, an onset to the exothermic action at about 250°C, and a peak at 261°C, at a heating rate of 3°C/min.

DSC data from both NWC and Hercules sources are summarized in Table 2. These DSC data are plotted in Figure 14 for GBP-1 and GCV. A plot of FKM data from a prior study is included as a reference point. The DSC data on QBC, UTP-15908A, KAA-114 and SAO-109 are plotted in Figure 15. The DSC data in Figures 14 and 15 are plotted as log ϕ/K^2 versus 1/K, as derived from the verified in open literature.[3,4] In these figures, the heating rate is related to the temperature of the maximum reaction. (The calculations used in this data treatment have been given in the report of footnote 2.) The data plots in Figure 14 indicate that GCV is more thermally stable than GBP-1, which in turn is more thermally stable than FKM; that is, more thermally stable in the temperature range of this study. In Figure 15, the plots show that QBC and UTP-15908A are about the same in regard to thermal stability but that KAA-114 differs from SAO-109 in thermal stability. The SAO-109 appears to have two decomposition paths; one path (DTA data) has a very low activation energy value. This path is reported, but not used in any of the calculations.

The plots described above were used to determine kinetic parameters for the six candidate propellants. These parameters are given in Table 3.

PREDICTIVE TECHNIQUES

INPUT DATA

Useful parameters were obtained from various sources for input into the heat flow equations. These sources are described in the following paragraphs.

[3] H. E. Kissinger. "Reaction Kinetics in Differential Thermal Analysis," *Anal. Chem.*, Vol. 29 (1957), p. 1702.

[4] K. Akita and M. Kase. "Relationship Between the DTA Peak and the Maximum Reaction Rate," *J. Phys. Chem.*, Vol. 72, No. 3 (1968), p. 906.

TABLE 2. DSC Data on Six Candidate Propellants.[a]

ϕ, K/min	Peak, T K	$1/K \times 10^3$	$\phi/°T^2 \times 10^7$	Source
GBP-1 Propellant				
5	453	2.207	4.1	Hercules
10	465	2.105	7.7	Hercules
40	491	2.036	27.6	Hercules
40	488	2.049	28.0	NWC
GCV Propellant				
3	466	2.46	2.3	DTA/NWC
10	482	2.075	7.2	NWC
20	489	2.045	13.9	NWC
40	499	2.004	26.8	NWC
QBC Propellant				
3	521	1.919	1.8	DTA/NWC
5	526	1.901	3.0	NWC
5	530	1.887	3.0	Hercules
10	532	1.880	5.9	NWC
40	543	1.841	22.6	NWC
UTP-15908A Propellant				
3	516	1.938	1.9	DTA/NWC
5	525	1.905	3.0	NWC
5	528	1.894	3.0	Hercules
10	531	1.883	5.9	NWC
10	532	1.880	5.9	Hercules
10	536	1.866	5.8	DTA/NWC
40	542	1.845	22.7	NWC
KAA-114 Propellant				
3	519	1.927	1.9	DTA/NWC
5	523	1.912	3.0	Hercules
10	543	1.842	5.7	DTA/NWC
40	555	1.802	21.6	NWC
SAO-109 Propellant				
3	535	1.879	1.7	DTA/NWC
5	593	1.686	2.4	NWC
10	570	1.753	5.1	DTA/NWC
10	611	1.637	4.5	NWC
40	628	1.592	16.9	NWC

[a] Some DTA data included.

TABLE 3. Kinetic Parameters on Candidate Propellants.

Propellant	Activation energy (E), kcal/mole	Log frequency factor, sec^{-1}
GBP-1	23.1	8.79
GCV	36.3	14.63
QBC	60.6	23.17
UTP-15908A	59.0	22.63
KAA-114	35[a]	12.2[a]
SAO-109	16.0[b]	3.9[b]
	41.4[c]	12.86[c]

[a] Approximate value from scattered data.
[b] Approximate value of DTA data.
[c] Approximate value of DSC data.

1. The test results described in the previous section provided the kinetic parameters of Table 3; these were used, along with other parameters, to obtain critical temperature (a heat balance calculation).

2. Time-temperature data based on the hazard scenarios of interest were taken either from the Hercules work (footnote 1) or from cook-off studies done previously at NWC.[5] In the footnote 1 report, Hercules developed time-distance-temperature plots that involved rapid heating of missiles by impinging exhaust jet (jet engine or huffer/jet engine starter). The time-temperature plots shown in Figure 16 for the propellant/liner interface temperature were approximated from Figures 8 and 9 of footnote 1 at three temperature levels: 400, 700, and 1000°F (at an air velocity of 450 ft/sec). The time-temperature plot for the fuel fire, as shown in the footnote 5 report for baseline propellant motor test No. 1, was determined to average 1500°F, and the case/liner temperature was taken at the inside wall of the motor tube prior to cook-off at 66 seconds.

3. Steady-state temperature versus distance data are shown in Figure 17. Exhaust temperature data for a huffer/jet engine starter (the MD-3B) were taken from Figure 4 of footnote 1 and for a turbojet engine (the J-52-P408) at intermediate thrust, from Figure 15 of footnote 1. The steady-state temperature value for a given heat source, as shown in Figure 17, is intended for use as a worst case with the four selected temperatures of Figure 16.

AUTOIGNITION TEMPERATURE CALCULATIONS

Equations for the critical temperature and the predicted time to cook-off at some surface temperature that exceeds the critical temperature can be obtained from

[5] Naval Weapons Center. *Evaluation of Concepts Applicable to Thermal Protections of In-Fleet Missiles*, by Jack M. Pakulak, Jr. and Carl M. Anderson. China Lake, Calif., NWC, November 1977, 216 pp. (NWC TM 3299, publication UNCLASSIFIED.)

footnote 2. Figures 14 and 15 are based on Equation 1, which can be used for simple autoignition temperature calculations where the propellant surface is heated very quickly and ignition occurs during this rapid heating.

$$\left(\frac{\phi}{T^2}\right)\left(\frac{E}{R}\right) = A\ \exp(-E/RT) \tag{1}$$

where

A = frequency factor
E = activation energy

For lower temperature heating rates, where self-heating can take place, the critical temperature, T_m, can be calculated from

$$T_m = \frac{E}{2.303\ R\ \log\left(\frac{a^2 \rho AQE}{\lambda R T_m^2 \delta}\right)} \tag{2}$$

For a surface temperature, T_1, that exceeds T_m, the reduced time, τ, can be calculated from

$$\tau = \frac{a^2 \rho c}{\lambda} \tag{3}$$

For a given value of T_1, the time to cook-off, t_e, can be determined from the following equation in conjunction with Figure 18:

$$\frac{t_e}{\tau} = 1.134 - 0.1842E\left(\frac{1}{T_m} - \frac{1}{T_1}\right) \tag{4}$$

PREDICTION OF COOK-OFF TIMES

To simplify the prediction techniques used in this study, a model for a solid rocket motor was established with the following conditions:

radius	= a = 6.35 cm (5 in.) in diameter
heat capacity	= c = 0.3 cal/g
diffusivity	= λ = 0.0005 cal/cm-K
heat of reaction	= Q = 500 cal/g
density	= ρ = 1.7 g/cm^3
gas constant	= R = 1.987 cal/mole-K
empirical constant	= δ = 2.00 (solid cylinder)
	Kinetic parameters from Table 3 are assumed.

Data from Figures 14 and 15 are assumed for estimating ignition temperatures and cook-off times under rapid heating.

A time limit of 25 minutes is established.

EXAMPLE 1

The time to cook-off or ignition for a rocket motor can be estimated from Figure 14 or 15, Equation 1, and Figure 16 when the heating rate is very fast, as in a fuel fire. For example, using SAO-109(2) propellant data (Figure 15 and Equation 1), the estimated time to cook-off is about 1 minute. To make this calculation, the heating rate, ϕ, from the fuel fire was estimated as 9°F/sec to about 750°F, the estimated ignition temperature of SAO-109(2) at this heating rate. Using these guesses and the kinetic parameters of SAO-109(2) propellant, Equation 1 was balanced; and, using Figure 16 with the assumptions that the liner is very thin (noninfluencing), the time to cook-off was 0.98 minute at 750°F. This is about the time to cook-off of a Sidewinder motor in a fuel fire. Using the same conditions with GBP-1 propellant in this motor size would give a cook-off time of about 0.6 minute at 523°F. The difference in cook-off time between the most thermally stable and the least thermally stable propellant would be almost within the experimental error experienced during fuel fire testing.

EXAMPLE 2

The steady-state temperature level at 1000°F would represent a jet engine located about 18 feet from the rocket motor or a huffer at about 1.0 foot from the rocket motor. The SAO-109(2) propellant motor would cook-off in about 6 minutes at 757°F (Equation 1) and the GBP-1 propellant in about 1.5 minutes at 517°F (Equation 1). These predicted values were estimated from the peak temperatures obtained by DSC (shown in Figures 14 and 15).

EXAMPLE 3

The next steady-state temperature level at 700°F would represent a jet engine at about 26 feet or a huffer at about 2.2 feet from the rocket motor. At this temperature level, the propellant-case interface temperature would not reach the "ignition temperature" of SAO-109(2) propellant within 25 minutes. Using Equation 2, the critical temperature for SAO-109(2) was 377°F. Since the conditions at the propellant-case interface are not quite isothermal, warm-up time to an approximate surface temperature is used here for ease of calculation. A warm-up time of 2 minutes was needed to reach a surface temperature (T_1) of 533°F. The time to cook-off after reaching this surface temperature was 14 minutes using Equations 3 and 4 and Figure 18. The total time to cook-off would be about 2 + 14 = 16 minutes. This cook-off time is considerably shorter than the time estimated from "ignition temperature" alone. The GBP-1 propellant would cook-off from simple skin ignition in about 3 minutes.

EXAMPLE 4

At the steady-state temperature level of 400°F, the rocket motor with SAO-109(2) propellant does not reach the critical temperature of this propellant within 25 minutes. This temperature of 400°F would represent a jet engine at 48 feet or a huffer/engine starter at 8 feet from a rocket motor. Using the DSC peak temperature data and Equation 1, the GBP-1 propellant may or may not ignite in 25 minutes. Taking into account the self-heating effects, the critical temperature for the GBP-1 propellant was calculated from Equation 2 to be 146°F. Assuming a surface temperature of 343°F and a warm-up time of about 4 minutes, the time to cook off is 19 minutes, for a total time of about 23 minutes.

The GCV propellant should not ignite within 25 minutes, using the DSC data from Figure 14.

Calculating the critical temperature at 212°F for the GCV propellant and using the same warm-up time and surface temperature as for GBP-1 propellant, the time to cook-off is 12 minutes, for a total time of 4 + 12 = 16 minutes. The reasons for the shorter time to cook-off of GCV, as compared to GBP-1, are the described test conditions and the kinetic parameters. For the UTP-15908A propellant, the rocket motor would not reach the critical temperature, 330°F, prior to 25 minutes.

It should be pointed out that even if the heat source is removed from the rocket motor just prior to cook-off, the motor will still cook-off even though the cook-off time will be longer. This is referred to as the "point of no return". The conditions are known for this occurrence but were not included in this series of calculations.

CONCLUSIONS

In summary, this report dealt with the autoignition temperature of six candidate propellants and the self-heating prediction data used in calculating the time to cook-off. These topics were mentioned in the Hercules report (footnote 1), but no mention of time to cook-off was covered in their various plots of time-distance-temperature. Hercules used the autoignition temperature of a propellant as the limiting safety factor. For example, the following data were taken from Table II of footnote 1.

Propellant	Autoignition temperature, °F	Ability to withstand hazard from huffer for infinite time	
		Distance, feet	Temp., °F
(Assumed)	300	15	295
A	322	12.5	. . .
B	405	8	. . .
C	440	7	425
D	450	6.5	. . .
(Existing)	660	2.5	650

Propellant SAO-109(2) is similar to the "existing" propellant in regard to autoignition temperatures (Figure 15). Using the critical temperature for SAO-109(2) at 377°F and a surface temperature of 650°F, the time to cook-off, t_e, would be about 1 minute after warm-up for a 5-inch-diameter motor. This is one example which indicates that neglecting self-heating can be very misleading.

The methods and techniques given in this report can be used to predict the time and temperature for cook-off for any given propellant. The kinetic parameters used in this report were based on the initial exothermic peak of each propellant (DSC analysis) and the estimated overall heat evolved.

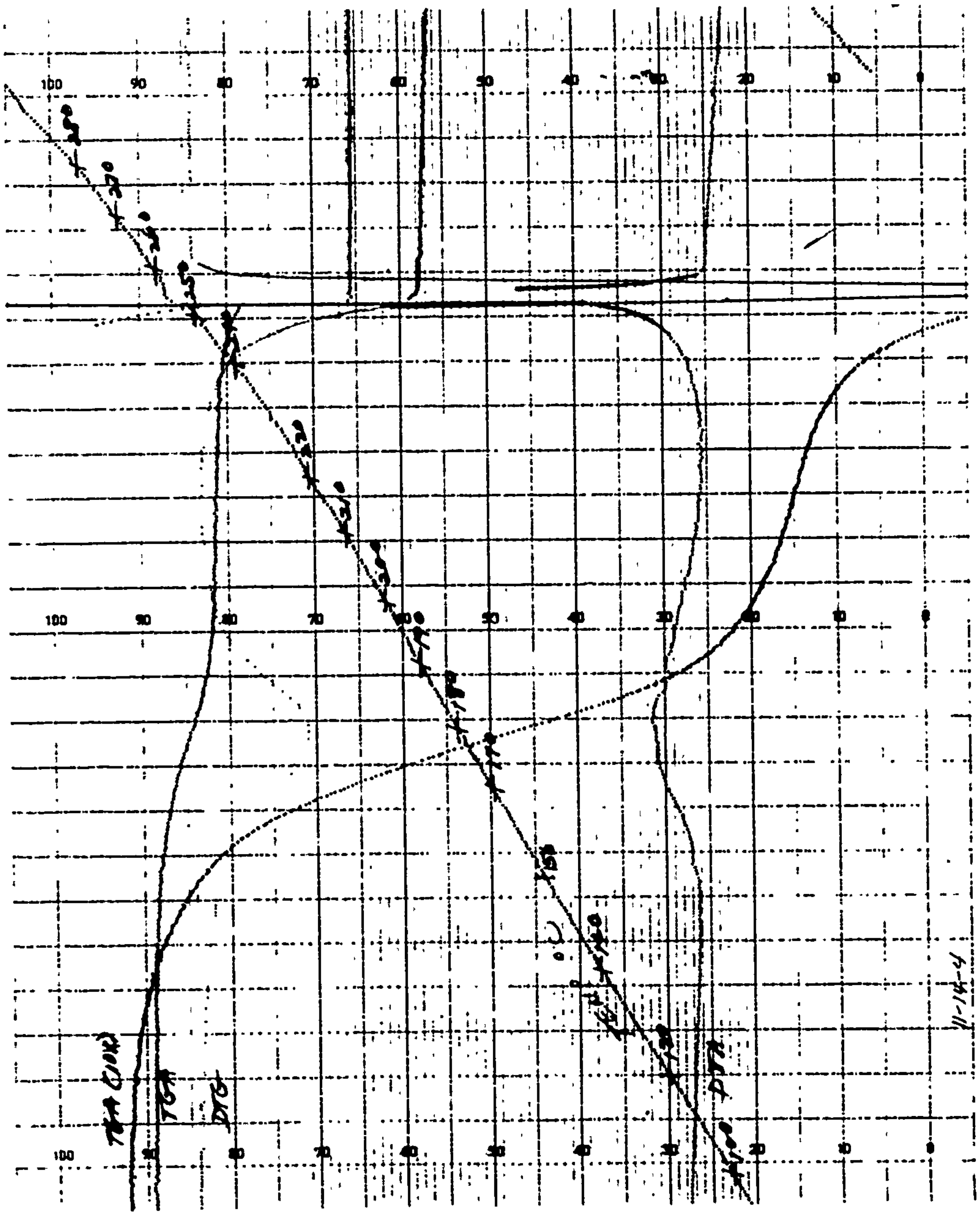

FIGURE 1. Thermal Patterns of GBP-1 at a Heating Rate of 3°C/Min. (Sample wt. = 25.045 mg; Run no. 11-14-4.)

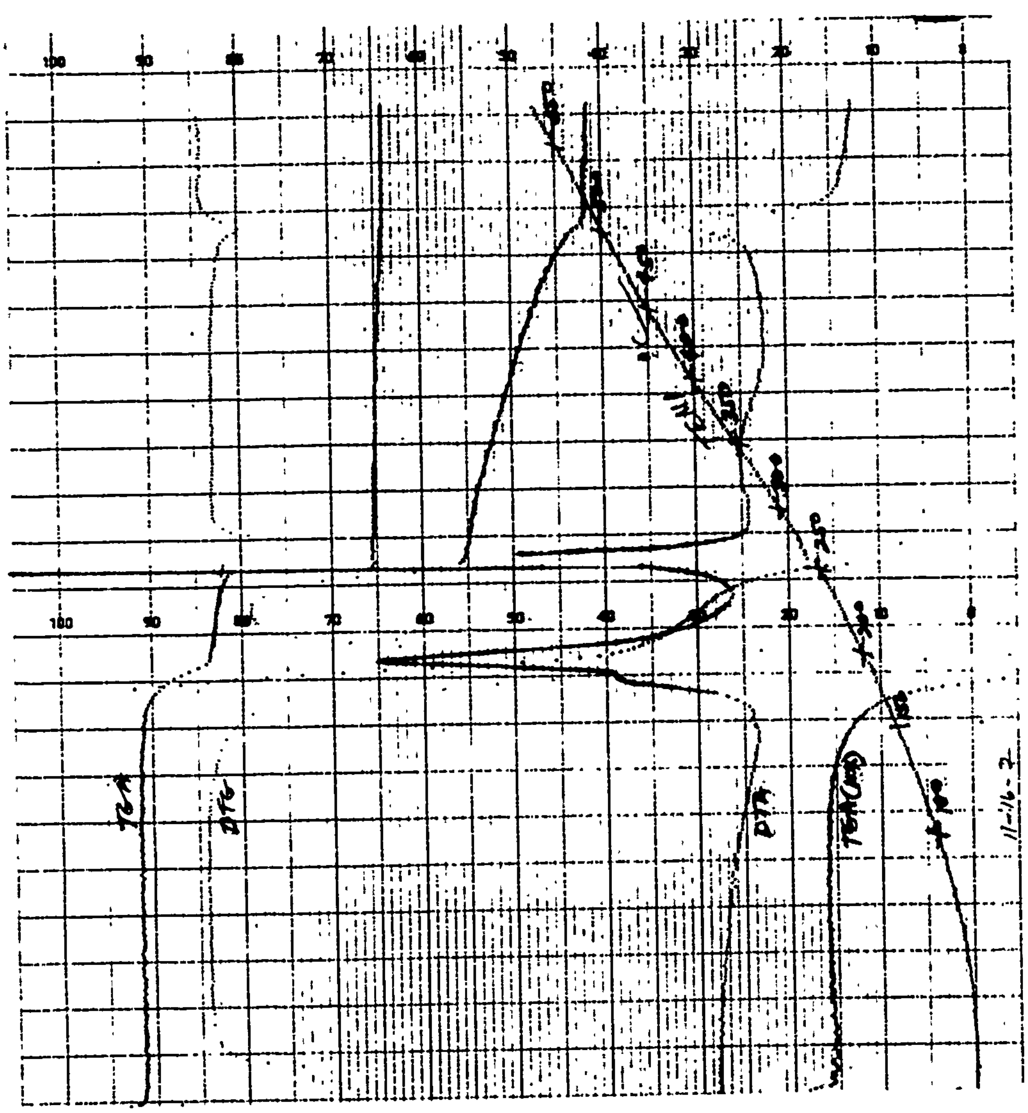

FIGURE 2. Thermal Patterns of GBP-1 at a Heating Rate of 10°C/Min.
(Sample wt. = 27.62 mg; Run no. 11-16-2.)

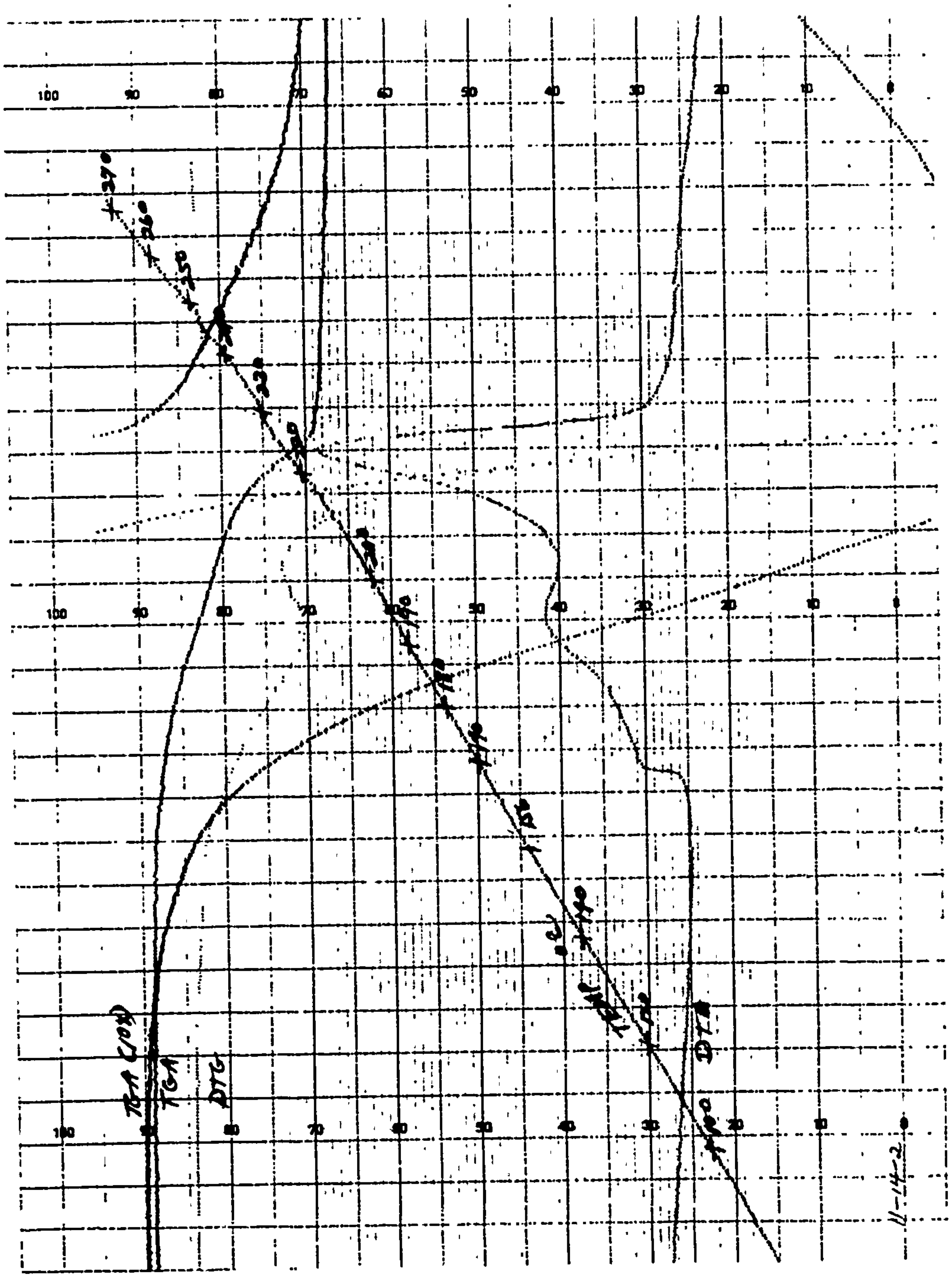

FIGURE 3. Thermal Patterns of GCV at a Heating Rate of 3°C/Min.
(Sample wt. = 24.15 mg; Run no. 11-14-2.)

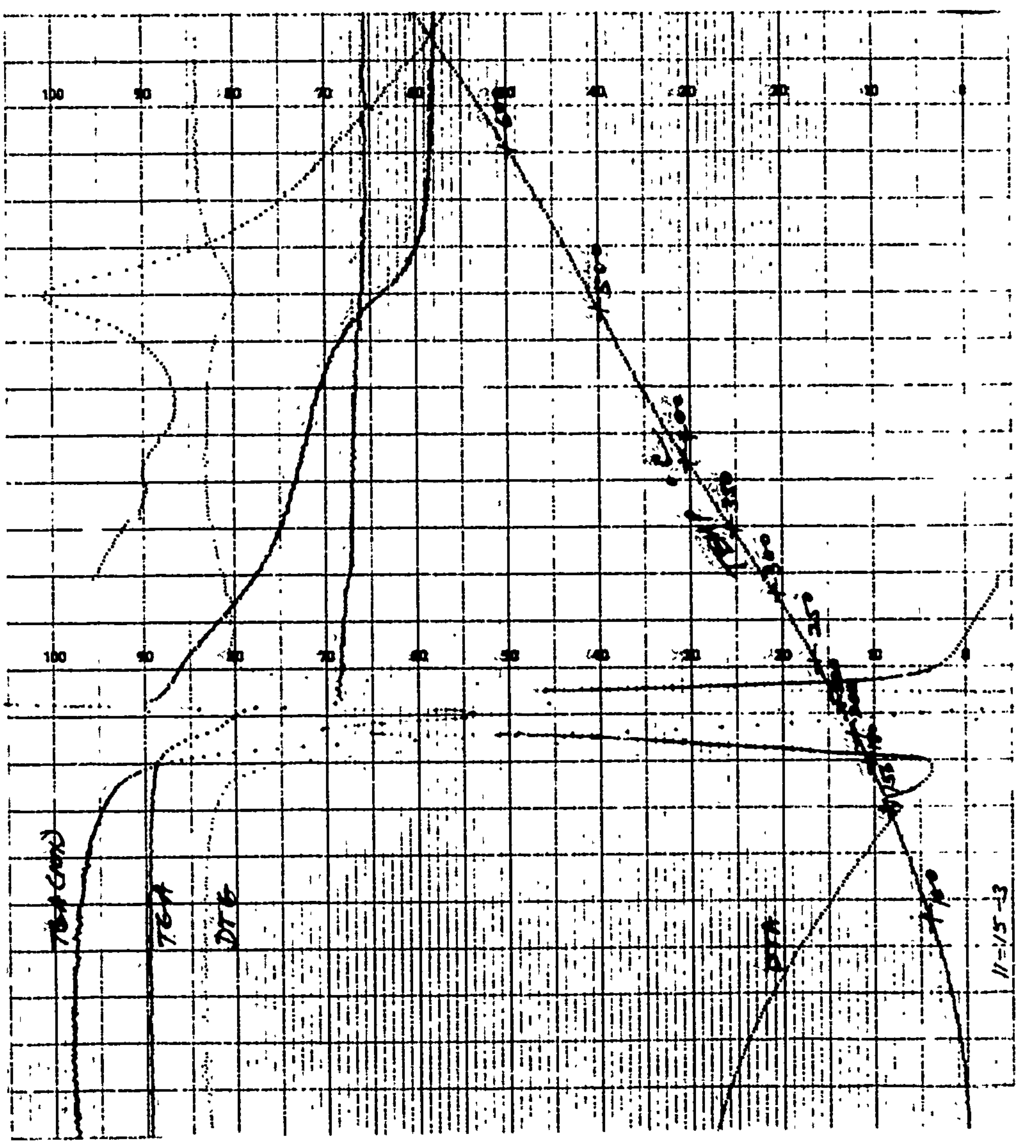

FIGURE 4. Thermal Patterns of GCV at a Heating Rate of 10°C/Min.
(Sample wt. = 24.00 mg; Run no. 11-15-3.)

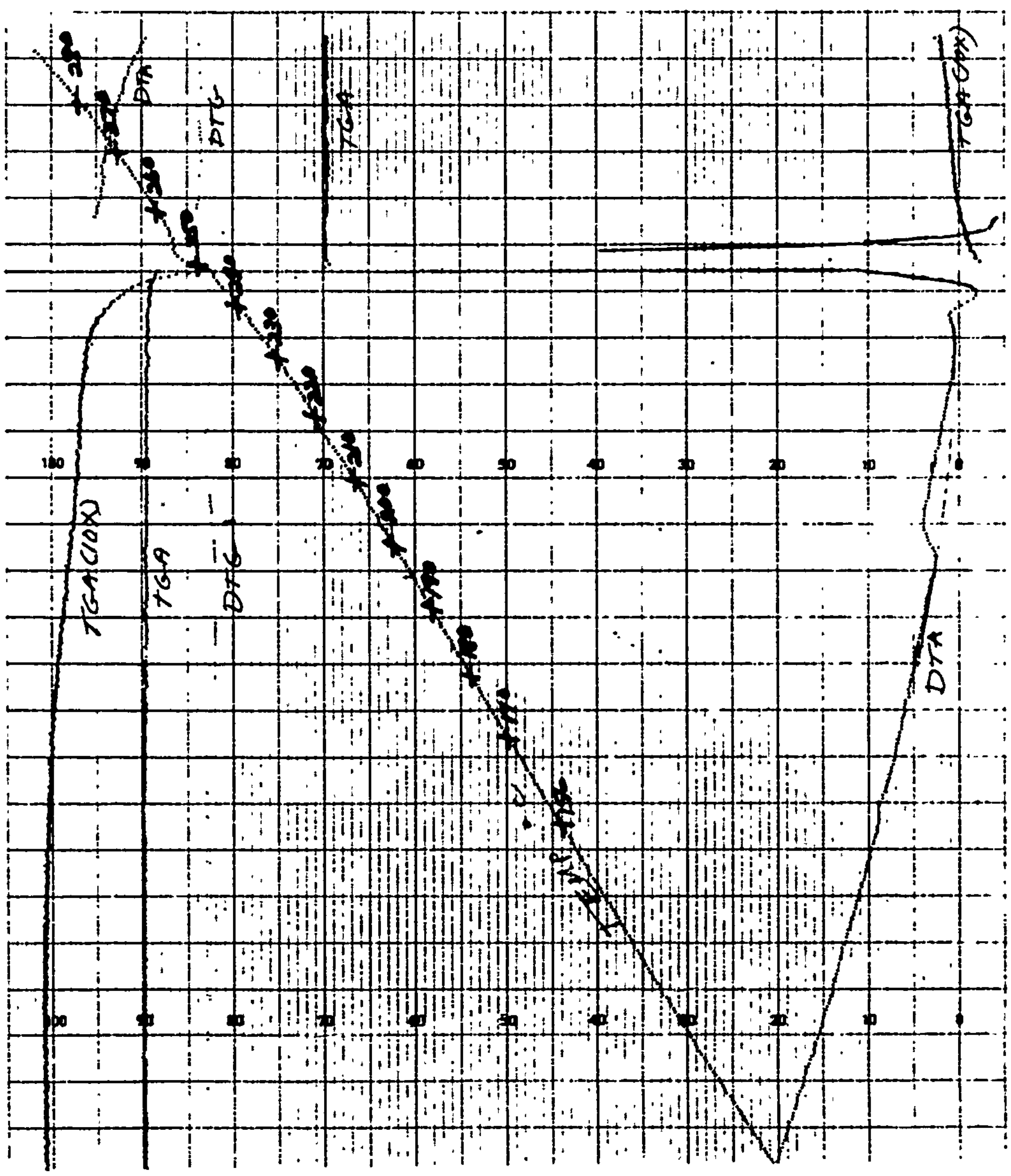

FIGURE 5. Thermal Patterns of QBC Propellant at a Heating Rate of 3°C/Min.
(Sample wt. = 23.253 mg; Run no. 11-24-2.)

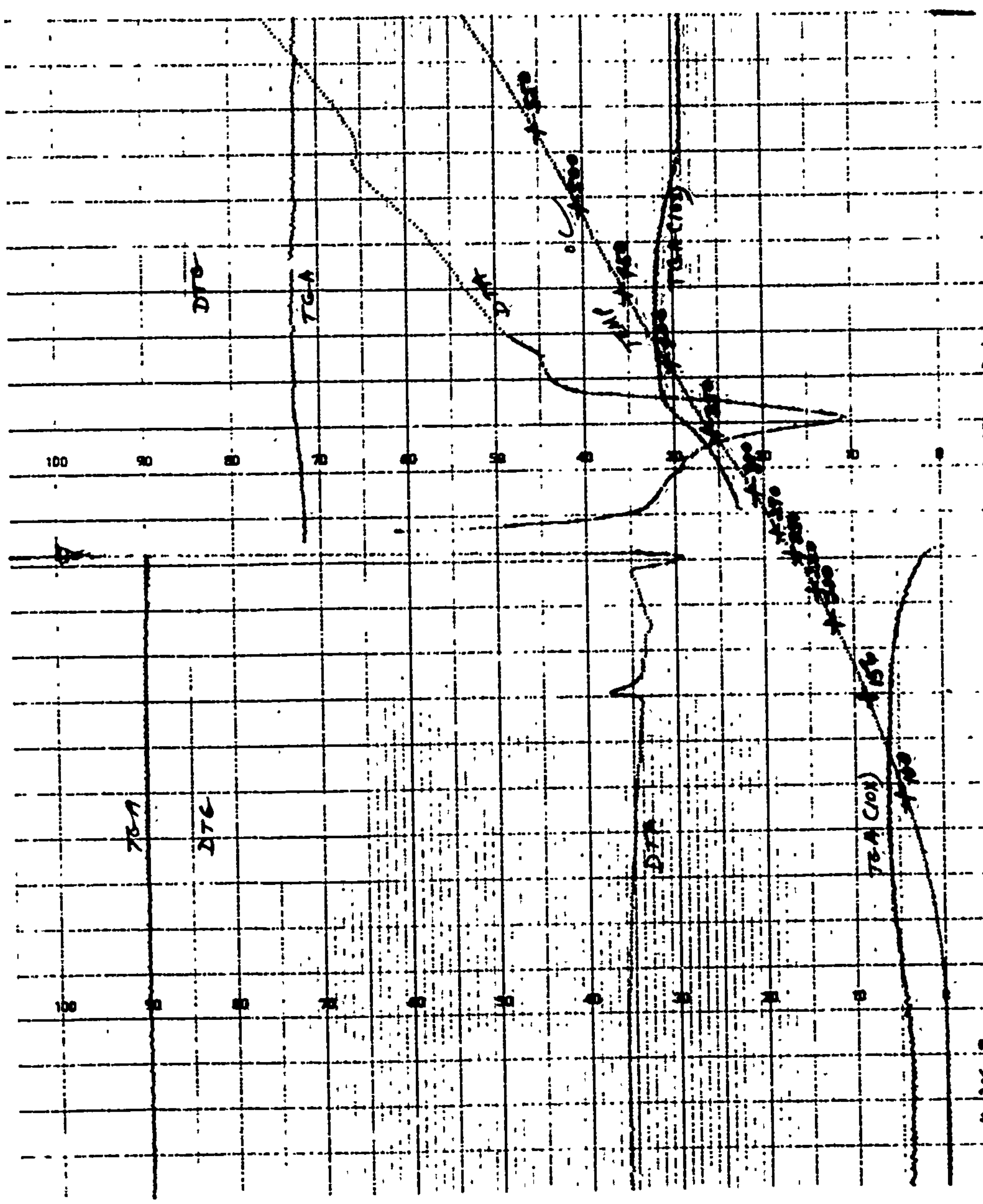

FIGURE 6. Thermal Patterns of QBC Propellant at a Heating Rate of 10°C/Min. (Sample wt. = 23.59 mg; Run no. 11-24-3.)

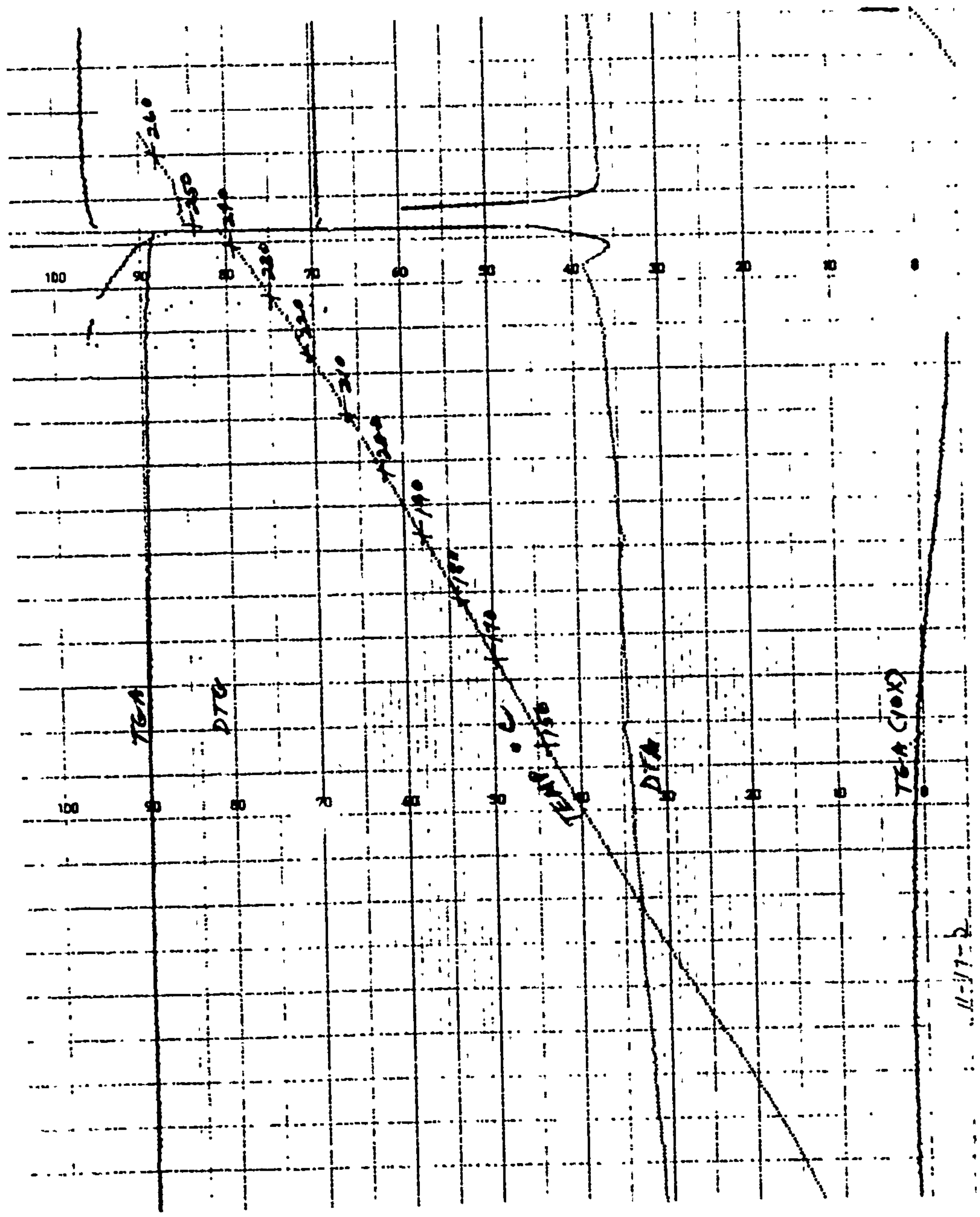

FIGURE 7. Thermal Patterns of UTP-15908A at a Heating Rate of 3°C/Min. (Sample wt. = 26.04 mg; Run no. 11-17-2.)

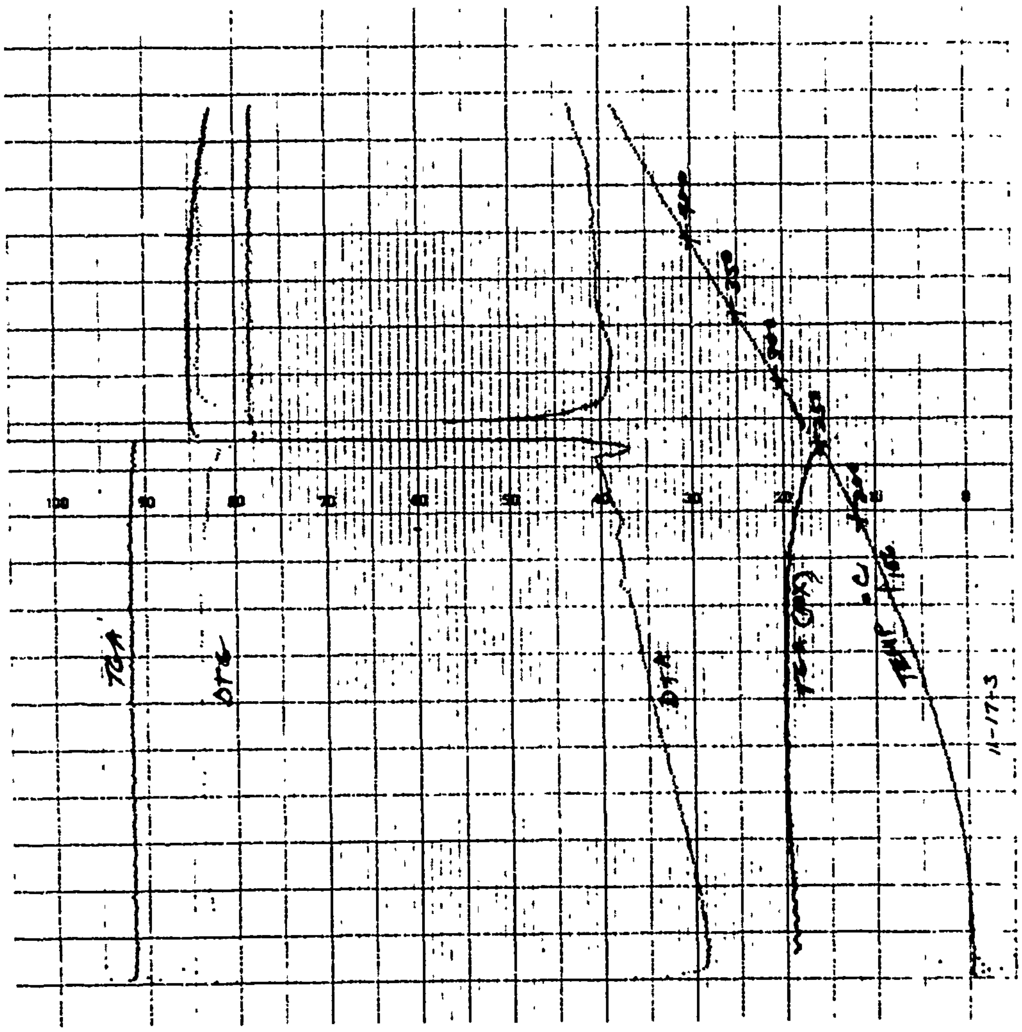

FIGURE 8. Thermal Patterns of UTP-15908A at a Heating Rate of 10°C/Min. (Sample wt. = 17.1 mg; Run no. 11-17-3.)

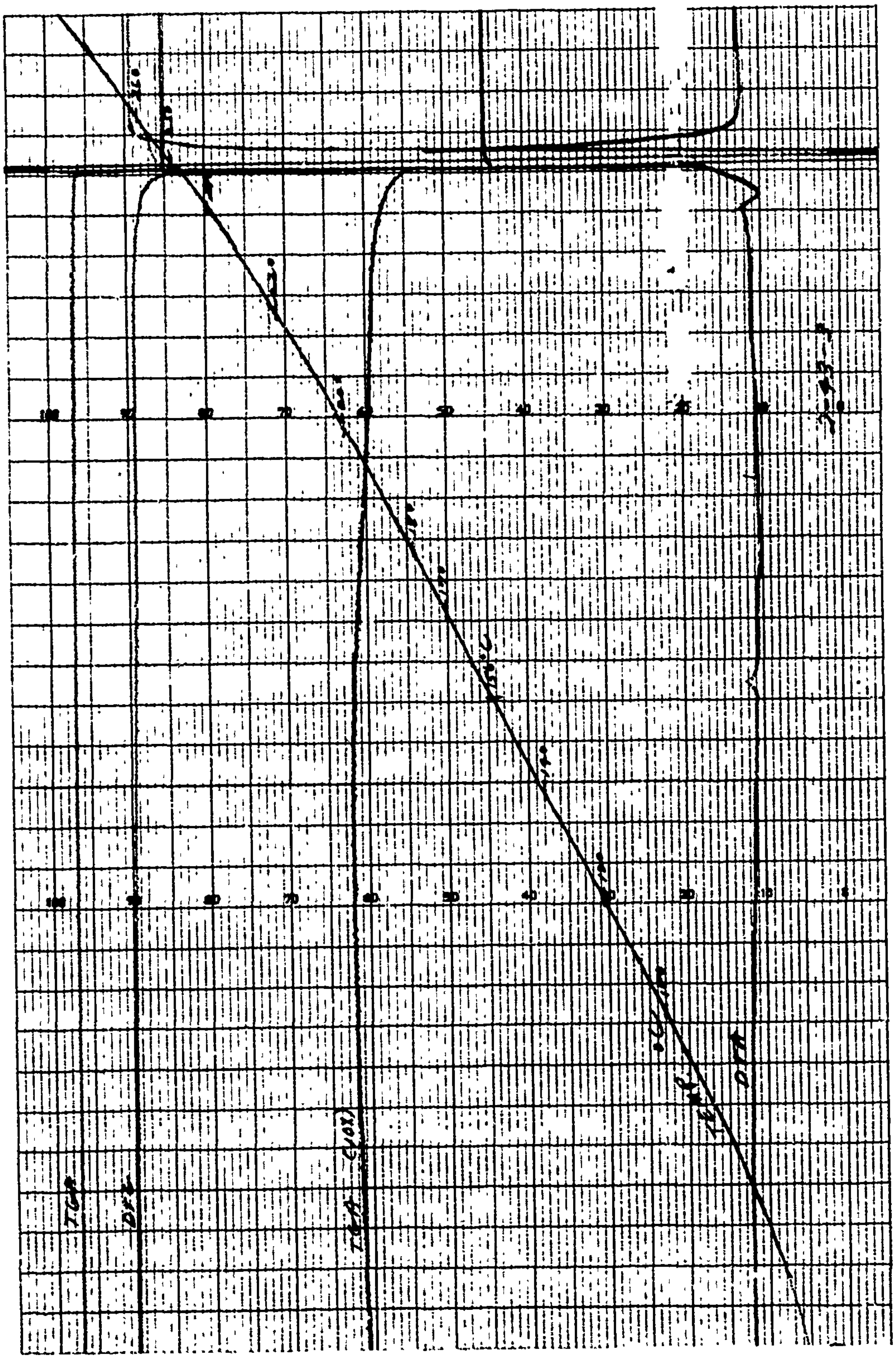

FIGURE 9. Thermal Patterns of Trident-Type Propellant Sample No. 7 (UTP-15908) at a Heating Rate of 3°C/Min. (Sample wt. = 15.1 mg; Run no. 2-43-3.)

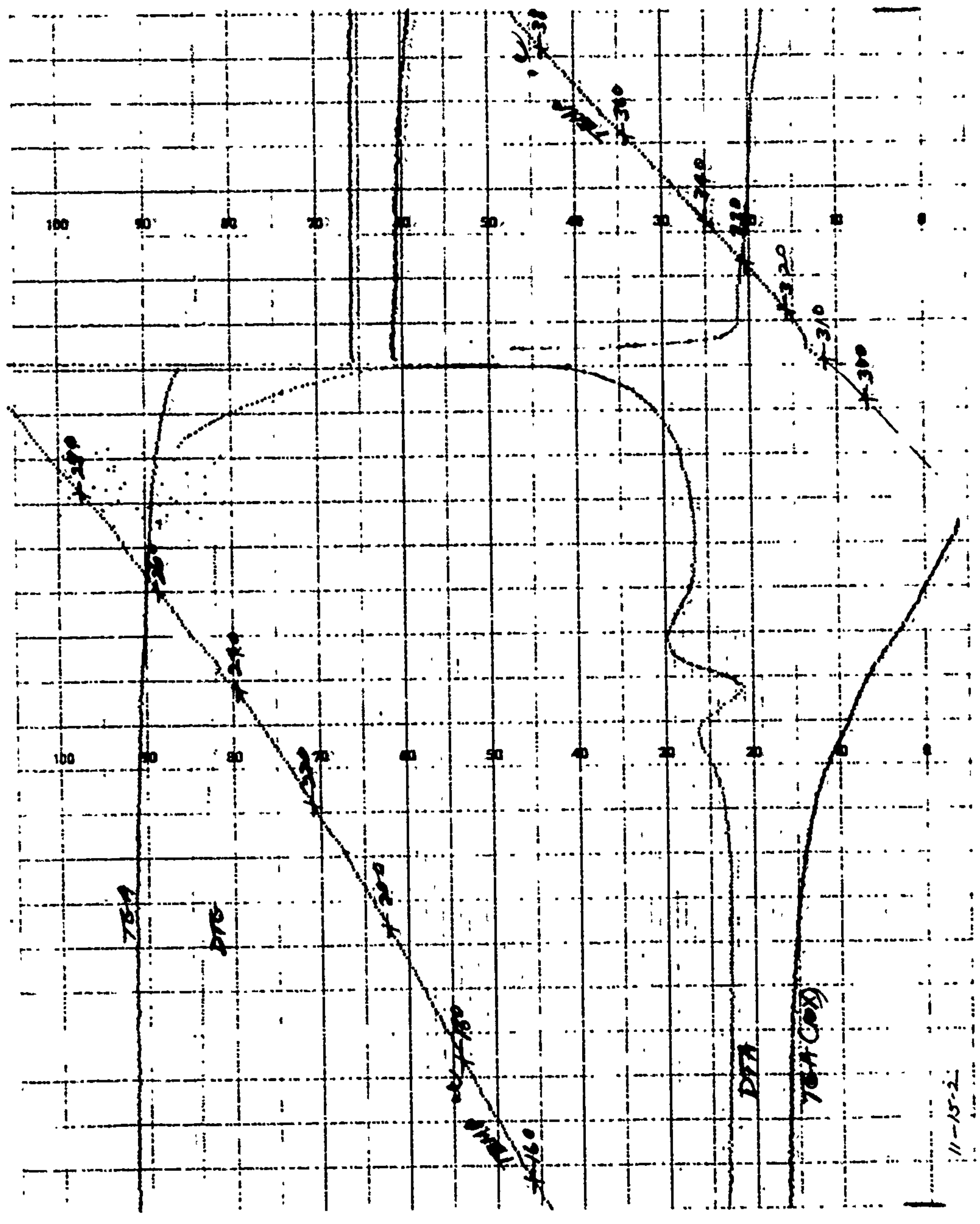

FIGURE 10. Thermal Patterns of KAA-114 at a Heating Rate of 3°C/Min.
(Sample wt. = 26.10 mg; Run no. 11-15-2.)

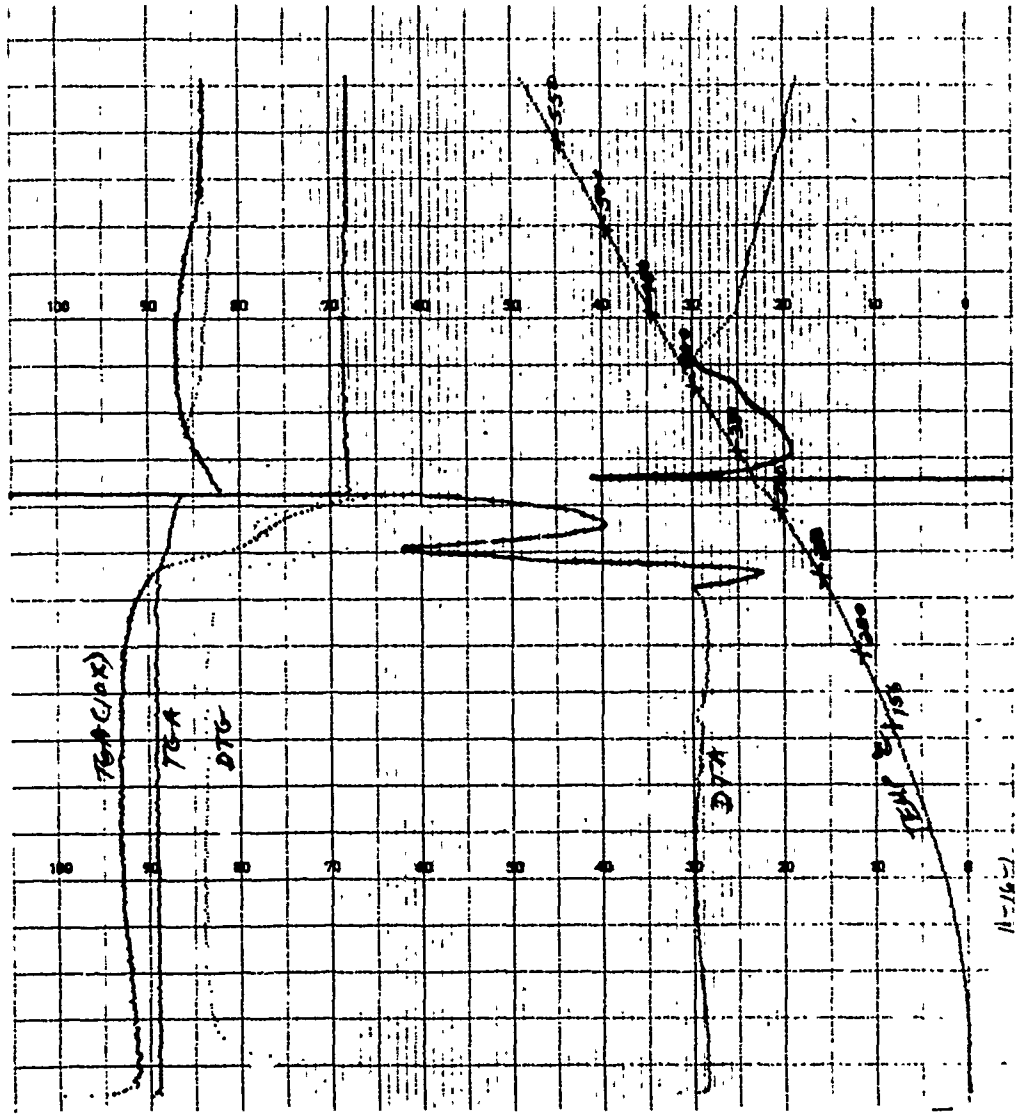

FIGURE 11. Thermal Patterns of KAA-114 at a Heating Rate of 10°C/Min.
(Sample wt. = 21.7 mg; Run no. 11-16-1.)

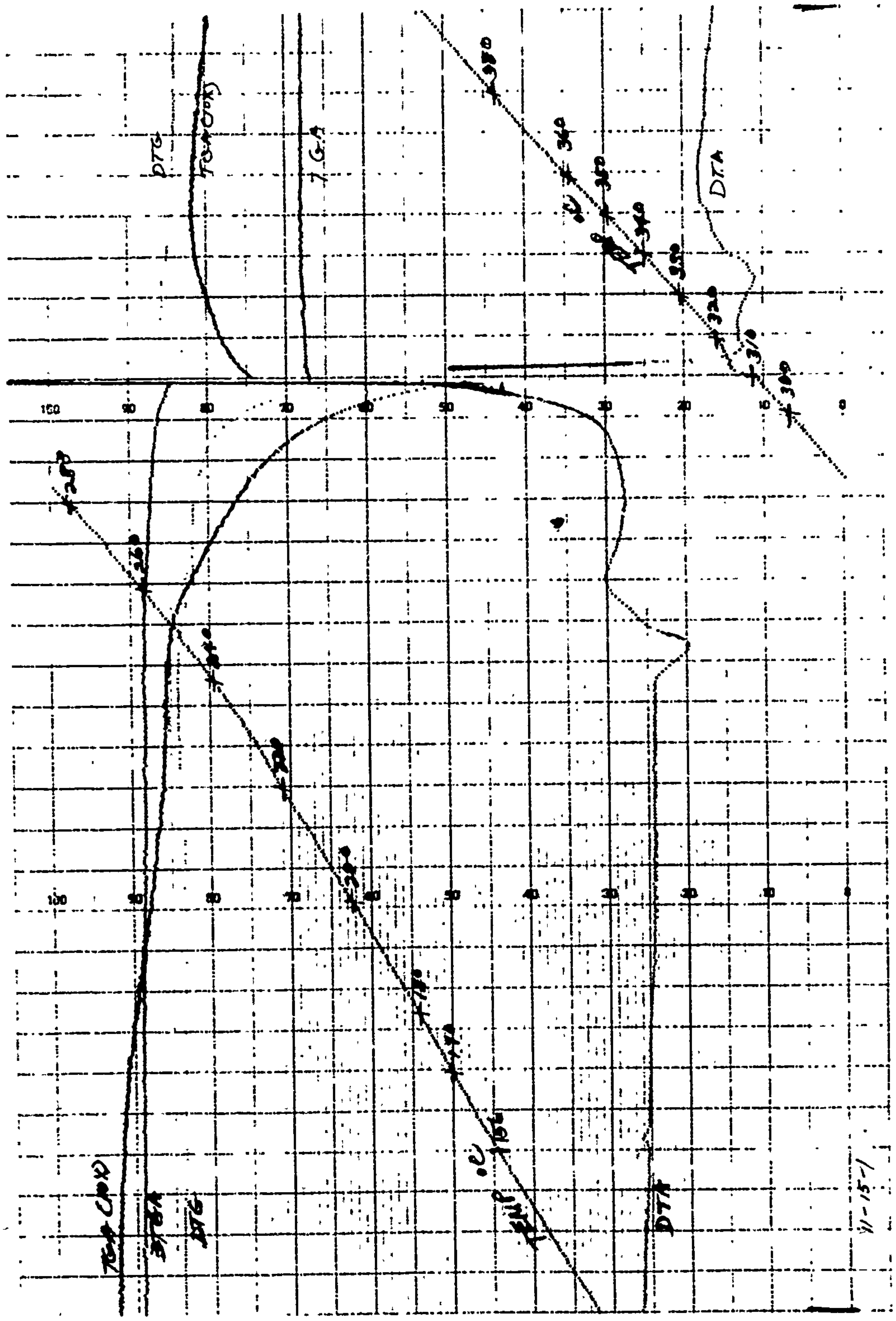

FIGURE 12. Thermal Patterns of SAO-109 at a Heating Rate of 3°C/Min. (Sample wt. = 22.2 mg; Run no. 11-15-1.)

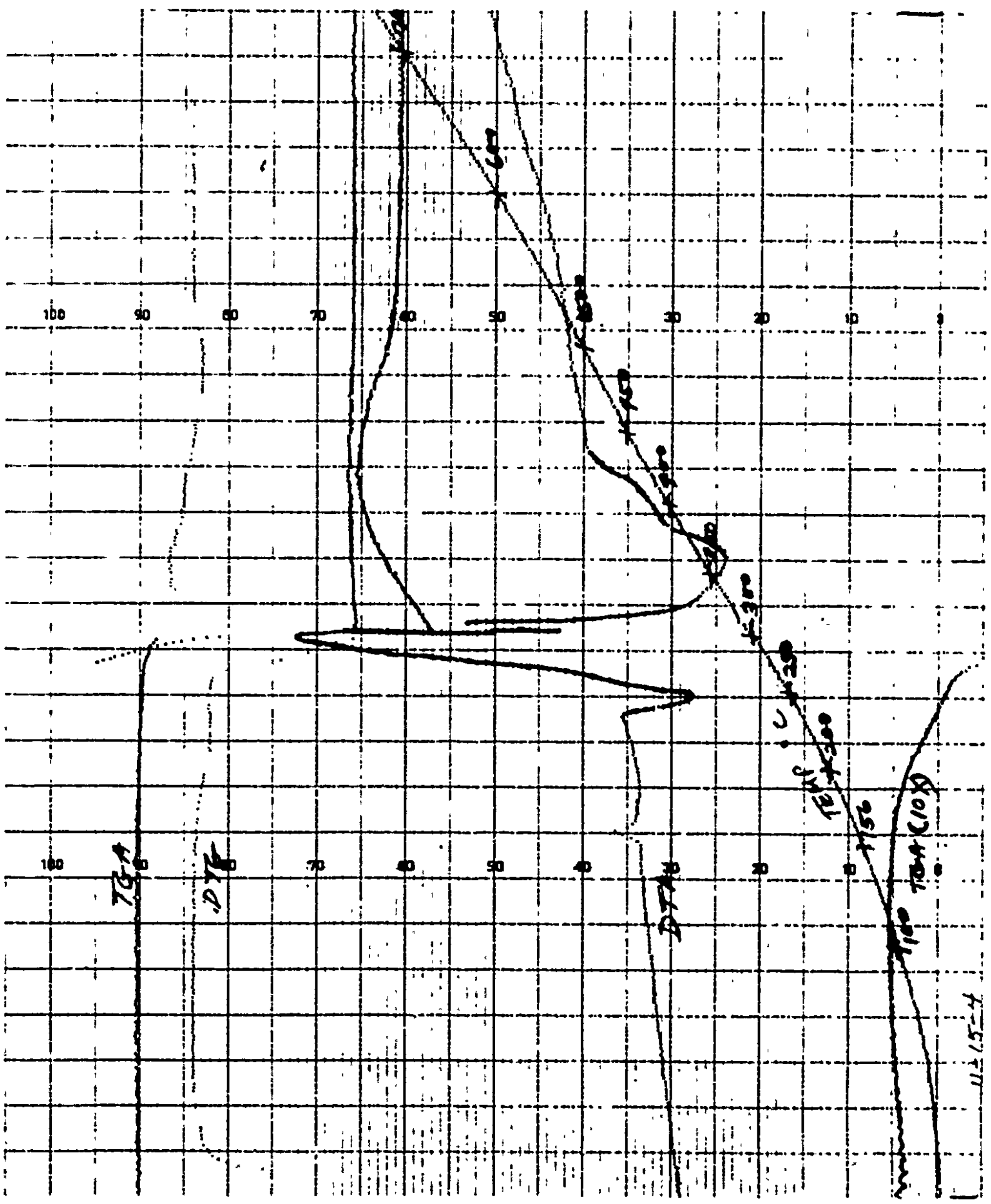

FIGURE 13. Thermal Patterns of SAO-109 at a Heating Rate of 10°C/Min.
(Sample wt. = 25.1 mg; Run no. 11-15-4.)

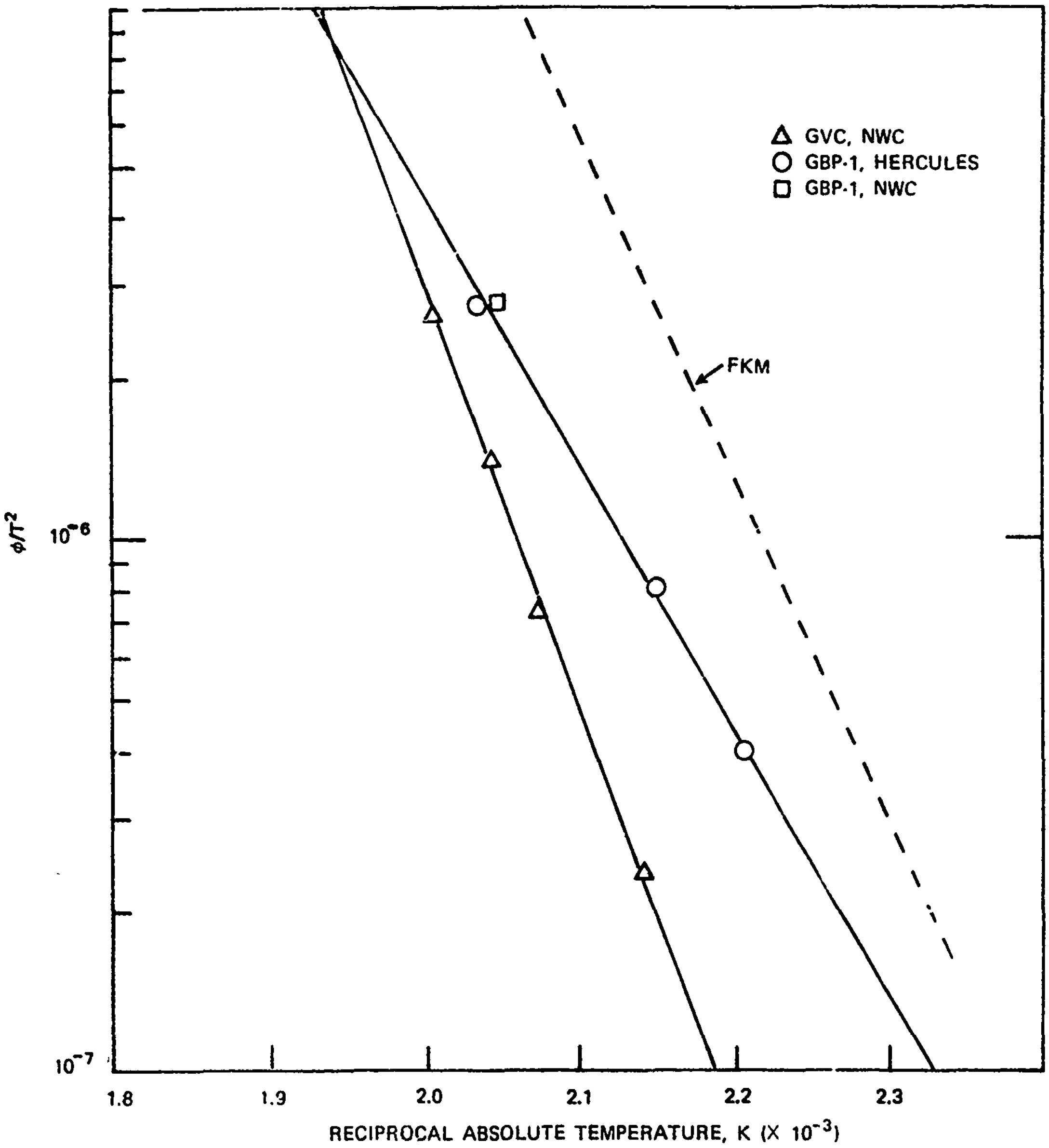

FIGURE 14. Plot of DSC Data on GCV and GBP-1 Propellants.

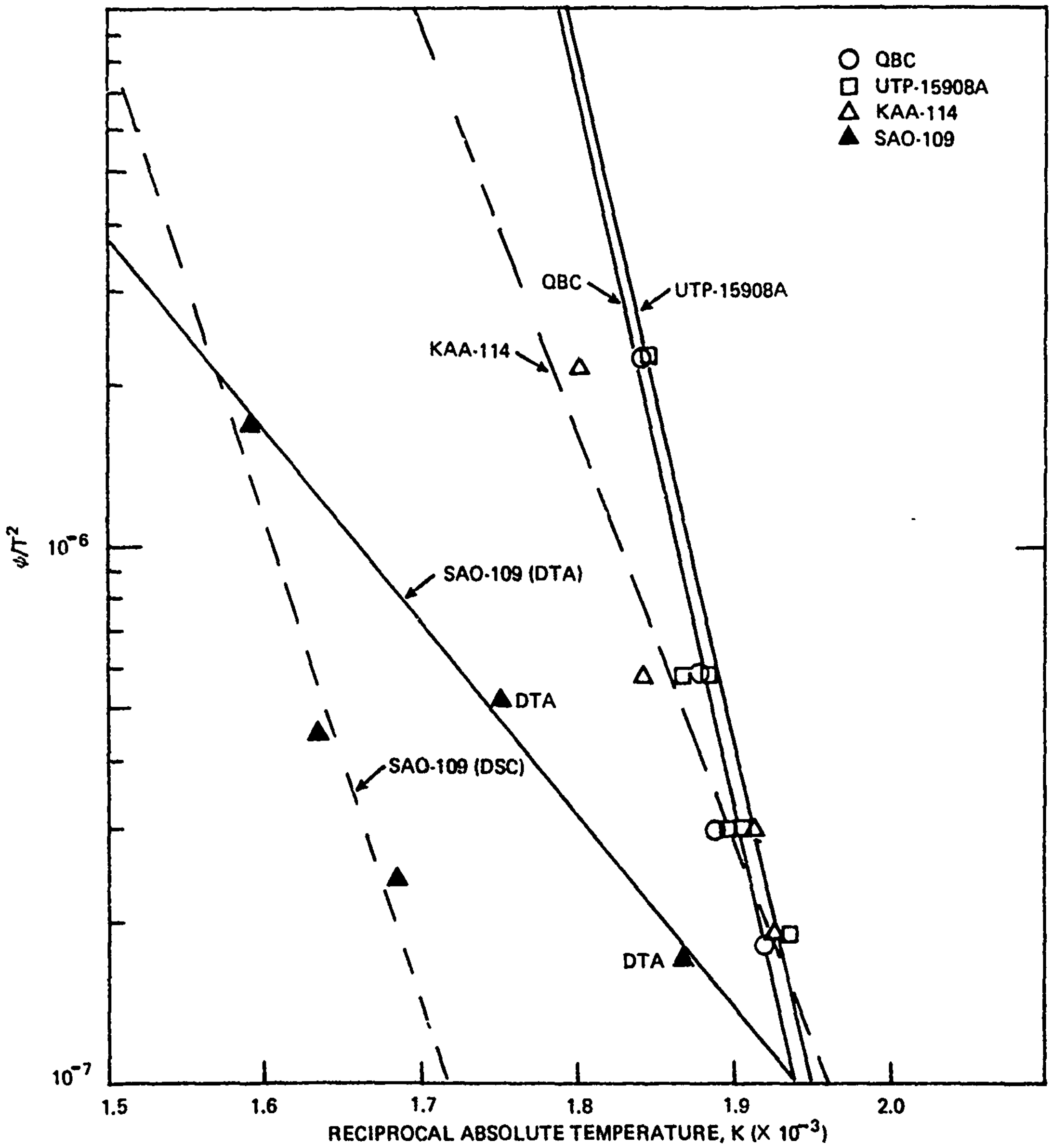

FIGURE 15. Plot of DSC Data on QBC, UTP-15908A, KAA-114 and SAO-109 Propellants.

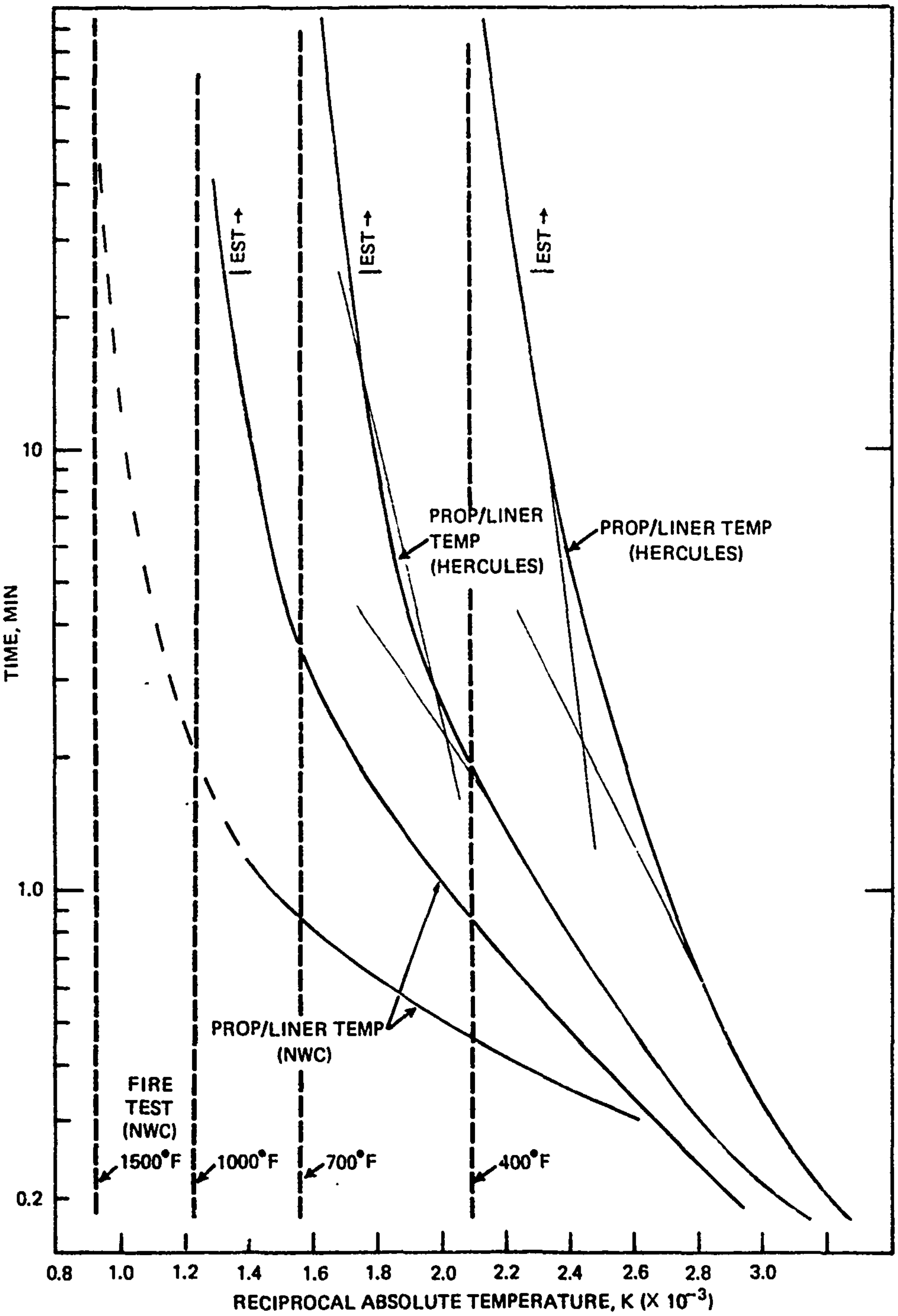

FIGURE 16. Time-Temperature Estimate Plots for Fuel Fire, Turbojet Engine, and Huffer Starter.

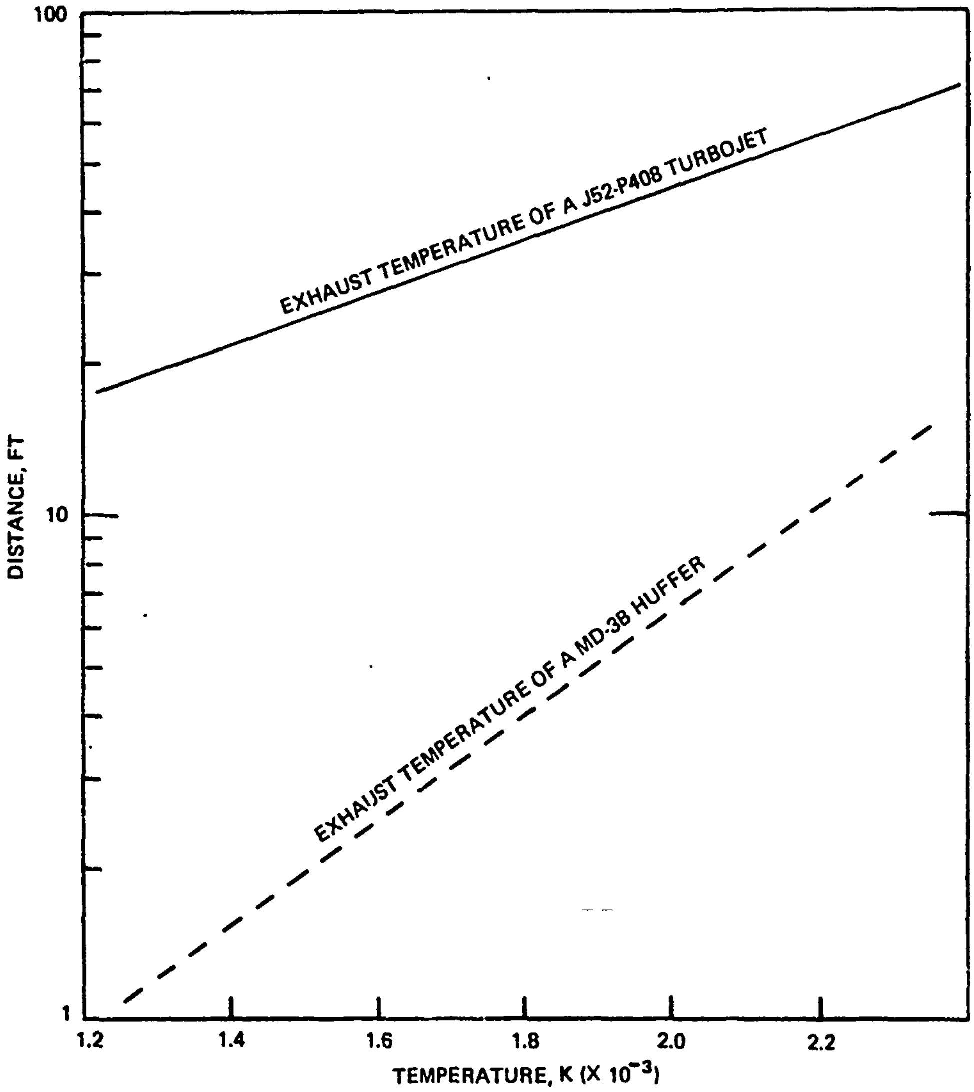

FIGURE 17. Plot of Distance vs. Temperature for Huffer and Jet Engine.

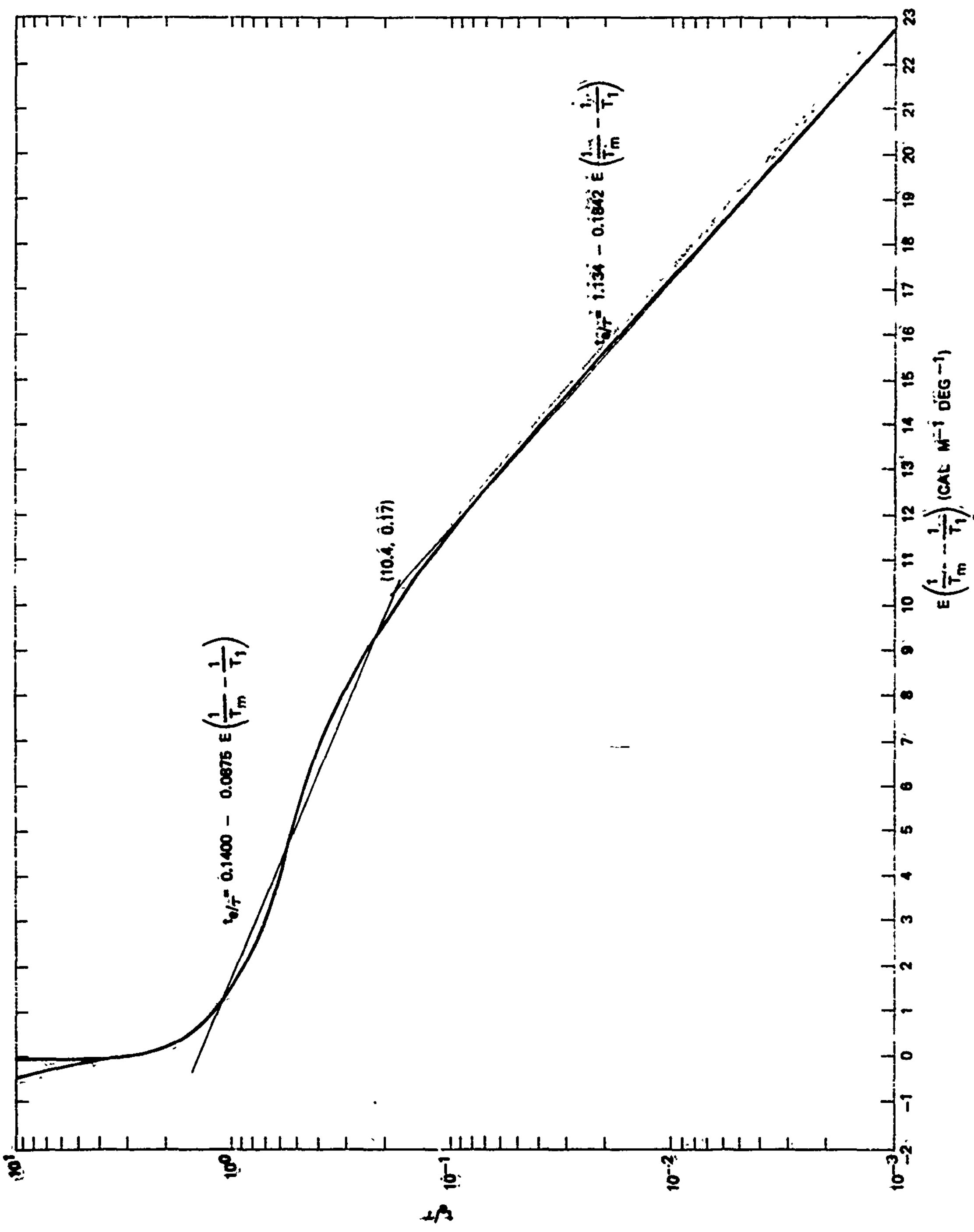

FIGURE 18. Approximation Equations for Time to Explosions.

NOMENCLATURE

a	Radius
A	Frequency factor
AP	Ammonium perchlorate
c	Heat capacity
CTPB	Carboxyl-terminated polybutadiene
DOA	Dioctyl adipate
DSC	Differential scanning calorimeter
DTA	Differential thermal analysis
DTG	Derivative thermogravimetry
E	Activation energy
FKM	Cross-linked composite modified double-base propellant
HMX	Cyclotetramethylenetetranitramine
HTPB	Hydroxyl-terminated polybutadiene
IDP	Isodecyl pelargonate
NG	Nitroglycerine
Q	Heat of reaction
R	Gas constant
RDX	Cyclotrimethylenetrinitramine
t_e	Time to cook-off
T_1	Surface temperature
TGA	Thermogravimetric analysis
T_m	Critical temperature
XLDB	Cross-linked double-base propellant
δ	Gas constant
λ	Diffusivity
ρ	Density
τ	Reduced time

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